

PROJECT NUMBER : 1752  
PROJECT TITLE : Molecular Structure Determination and Materials Evaluation  
PROJECT LEADER : G. Vilcins  
PERIOD COVERED : February, 1991

## **I. INSTRUMENTAL ANALYSES OF SIDESEAM ADHESIVE**

- A. **Objective:** To determine the proper instrumental procedure for the characterization of sideseam adhesives.
- B. **Results:** Four Fuller, four National, and one Ajax sideseam adhesives were analyzed by GC-FTIR, TC-MS, NMR, FTIR, MS, TGA, and X-Ray Fluorescence techniques. The results were evaluated to determine the procedures to be used for the characterization of the adhesive chemical composition.
- C. **Conclusions:** From these analyses it was determined that all four Fuller's adhesives, the AD-006 National, and the Ajax adhesives were formulated from polyvinyl acetate homopolymers. The other two National adhesives, AD-002 and AD-005, were formulated from polyethylene vinyl acetate copolymers. From this study it was concluded that for the adhesive chemical composition characterization only the NMR and FTIR instrumental analyses will be performed as they provided sufficient information on the adhesive chemical composition.
- D. **Plans:** No further work is planned at this time.
- E. **References:**
1. Bassfield, R., PM Notebook #7398, p. 179.
  2. Chung, C., PM Notebook #9035, pp. 24-28.
  3. Griff, M., PM Notebook #9042, pp. 19-20.
  4. Jensen, N., PM Notebook #8910, p. 85.
  5. Lyons-Hart, PM Notebook #7820, pp. 75-83.

## **II. METHOPRENE IN CARBON DIOXIDE**

- A. **Objective:** To determine the ability of liquid carbon dioxide to remove methoprene from tobacco.
- B. **Results:** Experiments were performed in which methoprene-treated tobacco was subjected to temperature, pressure, and CO<sub>2</sub> conditions in a model process. Methoprene levels in ground tobacco samples were reduced from 3.6±0.2 ppm to 1.5±0.3 ppm when the tobacco was emersed in liquid CO<sub>2</sub> (28 deg. F, 600 psi) for 30 minutes. In order to see if any methoprene could be recovered and positively

identified, 15 mgs of methoprene were added to a Whatman #40 filter paper and placed in a simulated impregnation vessel. A sample of 25 ml of liquid  $\text{CO}_2$  was pumped through the vessel and collected in 2-propanol. The 2-propanol was analyzed using supercritical fluid chromatography (SFC) with both flame ionization (FID) and infrared (FT-IR) detection. The FID on the SFC was used to quantify the amount of methoprene.

- C. **Plans:** Based on the results from the laboratory model system, further experiments will collect liquid  $\text{CO}_2$  from the DIET impregnator in the MC Primary.

D. **Reference:**

Thomas, E., Notebook #8492, pp. 65-66, 68-78.

### III. MS ANALYSIS OF $\text{SF}_6$

- A. **Objective:** To determine if  $\text{SF}_6$  is present in trace amounts in competitors expanded tobacco.
- B. **Results:** The requirements of the analysis for a highly specific method for a very volatile compound at trace levels from a complex mixture led to the development of methodology based on selected ion monitoring mass spectrometry. The study involved three major steps which included: 1) Determination of suitable ions and conditions for selected ion monitoring of  $\text{SF}_6$ . 2) Exploration of possible interfering species from the tobacco matrix. 3) Analysis of sample and suitable controls.
- C. **Conclusions:** The results show that  $\text{SF}_6$  can be detected at ppb levels in expanded tobacco. However, no  $\text{SF}_6$  was detected in the sample of interest.

D. **Reference:**

Jensen, N., "Mass Spectral Analysis of  $\text{SF}_6$ ," Memo to D. Leyden, February 3, 1991.